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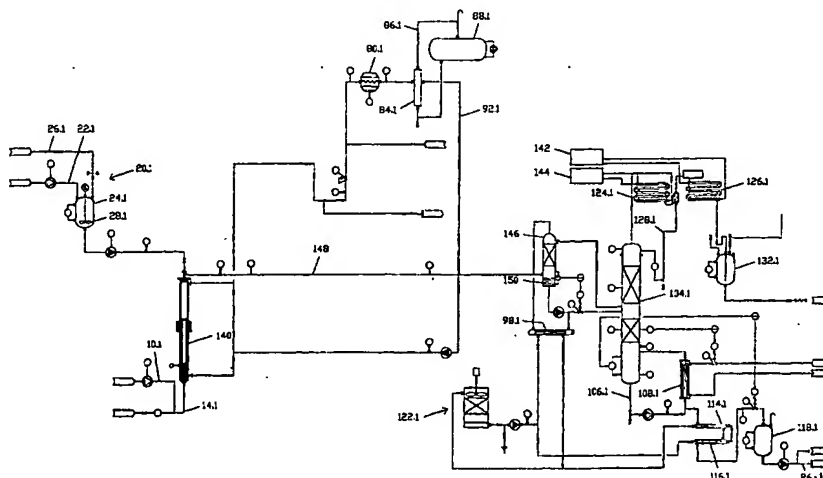
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(54) Title: THE MANUFACTURE OF CARBONYL SULPHIDE



(57) Abstract: A method of producing carbonyl disulphide is disclosed in which sulphur entering a vessel (24.1) along a line (22.1) is dissolved in liquid carbon disulphide entering along a line (26.1). In a reactor (140) the dissolved sulphur and carbon disulphide is reacted with carbon entering along a line (14.1) and solid carbon entering along a line (10.1). The reaction products are fed along a line (148) to a scrubber (146) and then to a distillation column (104.1). The gaseous fraction which emerges from the column (104.1), and which consists mainly of carbonyl sulphide, is cooled in two refrigeration stages (124.1, 126.1) and stored in a tank (132.1). Carbon disulphide is recovered from the column (104.1) and stored in a tank (118.1) for eventual return to the vessel (24.1) along the line (26.1).

THE MANUFACTURE OF CARBONYL SULPHIDEFIELD OF THE INVENTION

THIS INVENTION relates to the manufacture of carbonyl sulphide which is also known as carbonyl oxysulphide.

BACKGROUND TO THE INVENTION

Carbonyl sulphide, which is usually referred to as "COS", is being used for the fumigation of, for example, grain in silos. It is replacing earlier fumigants which are now considered to be ecologically unfriendly.

Commercially, COS is produced in one of two ways. One method involves reacting carbon monoxide with molten sulphur in a reaction vessel. The carbon monoxide is bubbled up through the molten sulphur. Molten sulphur is a dangerous, aggressive substance and hence specialized reaction vessels are required. Furthermore, carbon monoxide is a costly gas. As a consequence of these factors the resultant COS is expensive.

The second production method of which Applicant is aware comprises creating a heated fluidized bed of sulphur in particulate form and reacting it with the carbon monoxide. The sulphur is often in the form of pyrites so that the residue is iron which has a significant sulphur content and is hence not usable as feed stock for a smelting process.

-2-

Whilst this method results in a cheaper product than the first method described, the COS which is produced is still expensive.

COS, if produced at a lower cost, would be more widely used for fumigation than it is now and this has important ecological consequences.

Thus the present invention seeks to provide a process of COS production which results in COS that is less expensive than COS produced by the methods which are currently in commercial use.

#### BRIEF DESCRIPTION OF THE INVENTION

According to the present invention there is provided a process for producing carbonyl sulphide which comprises, in a first stage, dissolving sulphur in carbon disulphide and, in a second stage, reacting the solution of sulphur in carbon disulphide with a source of carbon and oxygen atoms.

The process can include the step of reacting the solution of sulphur in carbon disulphide with carbon and carbon dioxide. Alternatively the process can include reacting carbon dioxide and oxygen to provide carbon monoxide, and reacting the carbon monoxide with the solution of sulphur in carbon disulphide.

The carbon disulphide is separated from the carbonyl sulphide by, for example, feeding the carbonyl sulphide and the carbon disulphide to a distillation column.

-3-

The process can include the further step of cooling the carbonyl sulphide and the carbon disulphide carrier using a spray of liquid carbon disulphide and/or a bath of liquid carbon disulphide.

A specific form of the process includes the step of cooling the carbonyl sulphide and the carbon dioxide carrier using a spray of liquid carbon disulphide and/or a bath of liquid carbon disulphide, and feeding the carbonyl sulphide and the carbon disulphide contained therein from the cooling stage to a distillation column in which separation of carbon disulphide from carbonyl sulphide takes place.

The separated carbon disulphide can be recycled to said first stage. The process can further comprise the step of cooling the carbonyl sulphide emerging from the distillation column to produce a stream of carbonyl sulphide for refluxing to the distillation column and a stream of gaseous carbonyl sulphide.

The stream of gaseous carbonyl sulphide can, as a final process step, be liquified.

#### BRIEF DESCRIPTION OF THE DRAWINGS

For a better understanding of the present invention, and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings in which:-

Figures 1 and 2 illustrate a COS production plant;

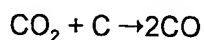
-4-

Figure 3 illustrates a detail of the plant of Figures 1 and 2; and

Figure 4 illustrates a further COS production plant.

#### DETAILED DESCRIPTION OF THE DRAWINGS

Referring firstly to Figure 1, carbon in the form of, for example, carbon black is fed by a screw conveyor along a line 10 to a reactor 12. Carbon dioxide is fed to the reactor 12 along a line 14. The reaction in the reactor 12 is:



A line 16 leads from the reactor 12 to a heat exchanger 18 in which the carbon monoxide produced is cooled.

Reference numeral 20 generally designates means for supplying sulphur dissolved in carbon disulphide. Sulphur, in solid form, is fed by a screw conveyor along a line 22 to a vessel 24 and liquid carbon disulphide is fed to the vessel along a line 26. The vessel is diagrammatically shown as having a paddle 28 for stirring the liquid carbon disulphide to cause the solid sulphur to dissolve in it. The liquid which flows out of the vessel 24 is at approximately 25°C.

A three stage reaction vessel 30, 32, 34 is provided for reacting the dissolved sulphur with the carbon monoxide produced in the reactor 12. The first stage reaction vessel 30 has an inlet 36 to which a line 38 leads from the heat exchanger 18. The first stage reaction vessel 30 has a further inlet 40 to which a

-5-

line 42 connects, the line 42 leading from the vessel 24 to the reaction vessel 30.

The arrangement of the inlets 36 and 40 is shown in Figure 3 which also illustrates an oil cooling jacket 44. The diameter of the flow path increases downstream of the inlet 40.

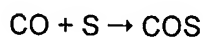
Pipes 48 and 50 connect the three stages of the reaction vessel to one another in series.

Liquid coolant is pumped into a cooling jacket 52 of the third stage 34 along a pipe 54. From the jacket 52 the coolant flows along a further pipe 56 to a jacket 58 of the second stage 32. A still further pipe 60 connects the jacket 52 to the jacket 44 (Figure 2).

From the stage 34 a mixture of gaseous COS and gaseous carbon disulphide flows to a further two stage reactor 62, 64 connected in series with one another by a pipe 66.

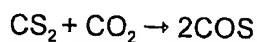
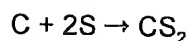
The stages 62, 64 are identical to the stages 30, 32, 34 and include jackets through which coolant is pumped. The coolant inlet pipe is designated 68.

The main reaction in the stages 30, 32, 34, 62, 64 is:



-6-

Subsidiary reactions are:



The liquid coolant circuit shown in Figure 1 includes the pipes 54, 56, 60 and 68. It also includes a pipe 70 which leads to the heat exchanger 18.

Return coolant pipes are shown at 72, 74 and 76. These join and continue as a single pipe 78 to a heat exchanger 80 which is used to dispose of excess heat. From the heat exchanger 80 a pipe 82 leads to a sump 84 in which solids settle out. A further pipe 86 leads from the sump 84 to a coolant storage tank 88 to which, on expansion of the coolant when it is heated, excess coolant flows.

An outlet pipe 92 leads from the sump 84 and constitutes a manifold which supplies coolant to the heat exchanger 18 and the reaction vessel stages 30, 32, 34, 62, 64.

The outlet of the last reactor stage 64 is designated 94 and a line 96 connected to it leads to a condenser 98 (see Figure 2). The condenser 98 has an outlet 100 for the gaseous fraction and an outlet 102 for the liquid fraction. The outlets 100 and 102 are connected to a distillation column 104.

In the column 104 the gaseous fraction, which consists mainly of COS,

-7-

risers and the liquid fraction, consisting mainly of liquid carbon disulphide, runs down the column 104.

The liquid which flows from the column 104 flows along a line 106 to a reboiler 108 fed with hot liquid along a pipe 110. The return heating liquid pipe is shown at 112. COS in the reheated liquid which is returned to the column 104 from the reboiler 108 rises in the column 104 and is recovered.

A liquid level detector is provided in the column 104. When this detects that the top surface of the carbon disulphide in the column 104 has reached a predetermined level, liquid carbon disulphide is diverted to a cooler 114, 116 before reaching a storage tank 118.

Carbon disulphide in the tank 118 is eventually returned to the line 26 by a pump 120 and recycled.

A cooling water circuit 122 including a heat dissipating radiator provides cooling water to the cooling stages 114, 116 and also provides chilled water for the condenser 98.

The gaseous fraction which flows out of the upper end of the column 104 comprises carbonyl sulphide and carbon disulphide and passes through two air refrigeration stages 124, 126. In the first stage cooling takes place to a temperature above that at which carbonyl sulphide liquifies but below that at which carbon

-8-

disulphide liquifies. The liquified carbon disulphide is returned to the column 104 along the line 128. In the second stage the carbonyl sulphide is cooled until it liquifies. From the second stage liquid COS is fed along a line 130 to a tank 132 and other carbon gases, such as carbon dioxide and carbon monoxide, are vented through an outlet 134.

Carbon dioxide cylinders are shown at 136 and 138 for supplying gas to the refrigeration stages 124, 126.

Turning now to Figure 4, the COS production plant illustrated has many features in common with the plant of Figures 1 and 2. Where applicable like references have been used with the addition of the suffix .1.

A reactor 140 comprises a carbon bed in a metal vessel lined with refractory material to withstand the heat of the exothermic reaction. The pipe 92.1 supplies coolant to the reactor. The reactions which, in Figures 1 and 2, take place in the reactor 12 and in the three stage reaction vessel 30, 32, 34 all take place in the reactor 140.

The refrigeration stages 124.1 and 124.2 are powered by two compressors designated 142, 144.

Between the reactor 140 and the column 104.1 there is a scrubber and cooler the vessel of which is designated 146. A line 148 connects the reactor 140 to

the vessel 146.

In the lower part of the vessel 146 there is a bath 150 of liquid carbon disulphide. This liquid is withdrawn from the vessel 146, pumped through a heat exchanger 98.1 and returned to the upper end of the vessel 146 as a spray of cooled liquid carbon disulphide.

The reaction products from the reactor 140 enter either above or below the liquid level in the vessel 146. If they enter below the liquid level, they first bubble up through the liquid and then flow countercurrent to the falling spray of liquid carbon disulphide. If the reaction products enter above the liquid level then they flow countercurrent to the falling spray.

Elemental sulphur in the reaction products is dissolved in the liquid carbon disulphide, and this prevents sulphur coatings building up in the column 104.1 and other parts of the plant.

-10-

CLAIMS:

1. A process for producing carbonyl sulphide which comprises, in a first stage, dissolving sulphur in carbon disulphide and, in a second stage, reacting the solution of sulphur in carbon disulphide with a source of carbon and oxygen atoms.
2. A process as claimed in claim 1, and including the step of reacting the solution of sulphur in carbon disulphide with carbon and carbon dioxide.
3. A process as claimed in claim 1, and including reacting carbon dioxide and oxygen to provide carbon monoxide, and reacting the carbon monoxide with the solution of sulphur in carbon disulphide.
4. A process as claimed in claim 1, 2 or 3 and including the further step of separating carbon disulphide from carbonyl sulphide.
5. A process as claimed in claim 4, and including the step of feeding the carbonyl sulphide and the carbon disulphide to a distillation column to separate carbon disulphide from carbonyl sulphide.
6. A process as claimed in claim 1, 2, 3 or 4 and including the step of cooling the carbonyl sulphide and the carbon disulphide carrier using a spray of liquid carbon disulphide and/or a bath of liquid carbon disulphide.

-11-

7. A process as claimed in claim 1, 2 or 3 and including the step of cooling the carbonyl sulphide and the carbon dioxide carrier using a spray of liquid carbon disulphide and/or a bath of liquid carbon disulphide, and feeding the carbonyl sulphide and the carbon disulphide contained therein from the cooling stage to a distillation column in which separation of carbon disulphide from carbonyl sulphide takes place.
8. A process as claimed in claim 4, 5, 6 or 7 and including the step of recycling separated carbon disulphide to said first stage.
9. A process as claimed in claim 7 and including the step of cooling the carbonyl sulphide emerging from the distillation column to produce a stream of carbonyl sulphide for refluxing to the distillation column and a stream of gaseous carbonyl sulphide.
10. A process as claimed in claim 9 and including the further step of liquefying the stream of gaseous carbonyl sulphide.

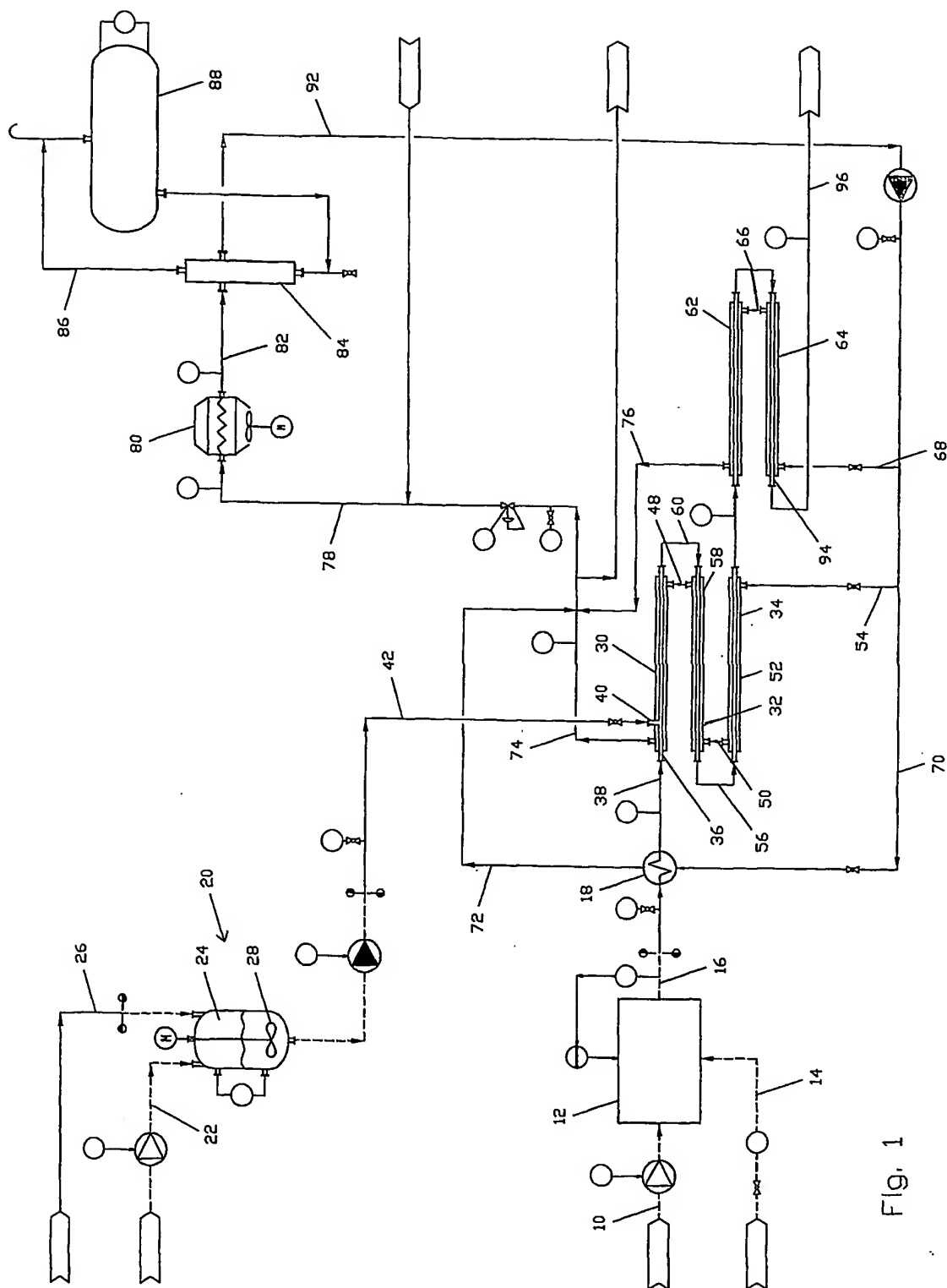


Fig. 1

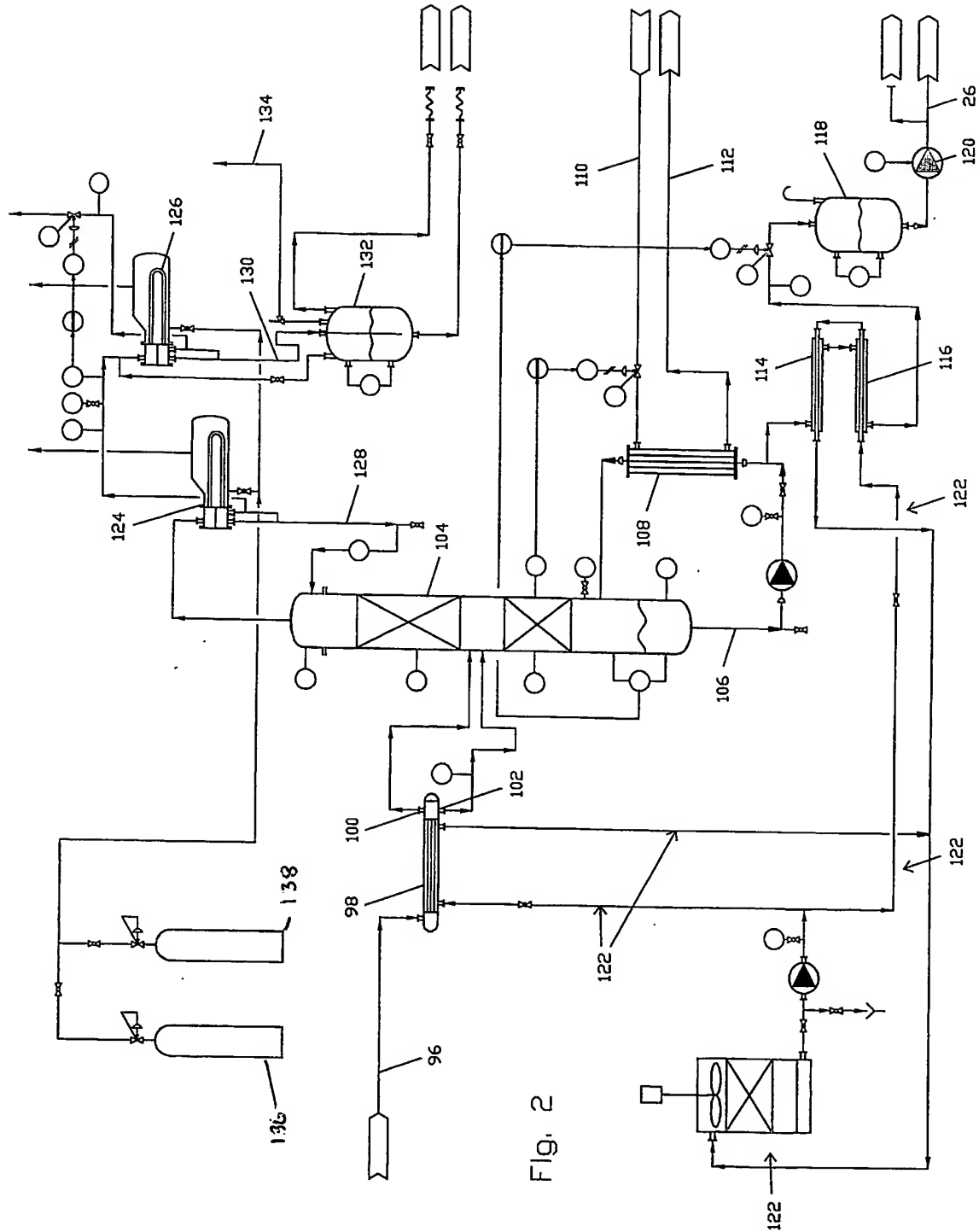


Fig. 2

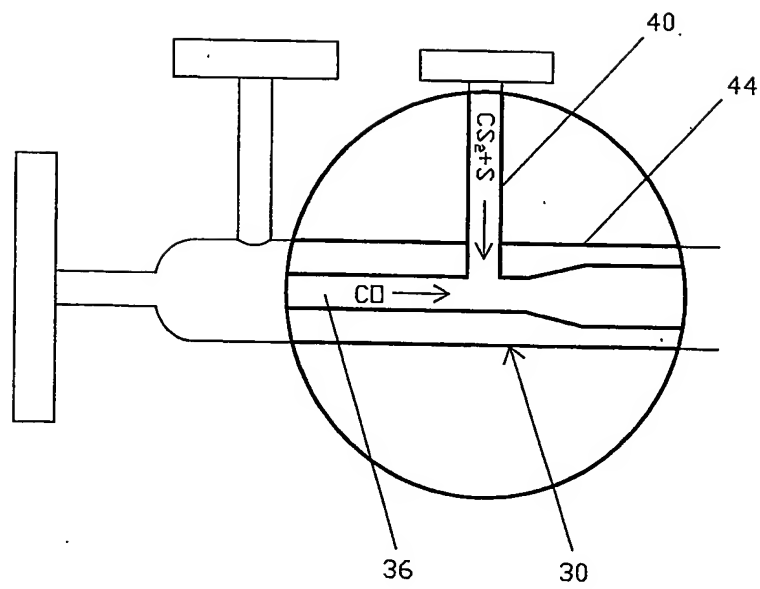


Fig. 3

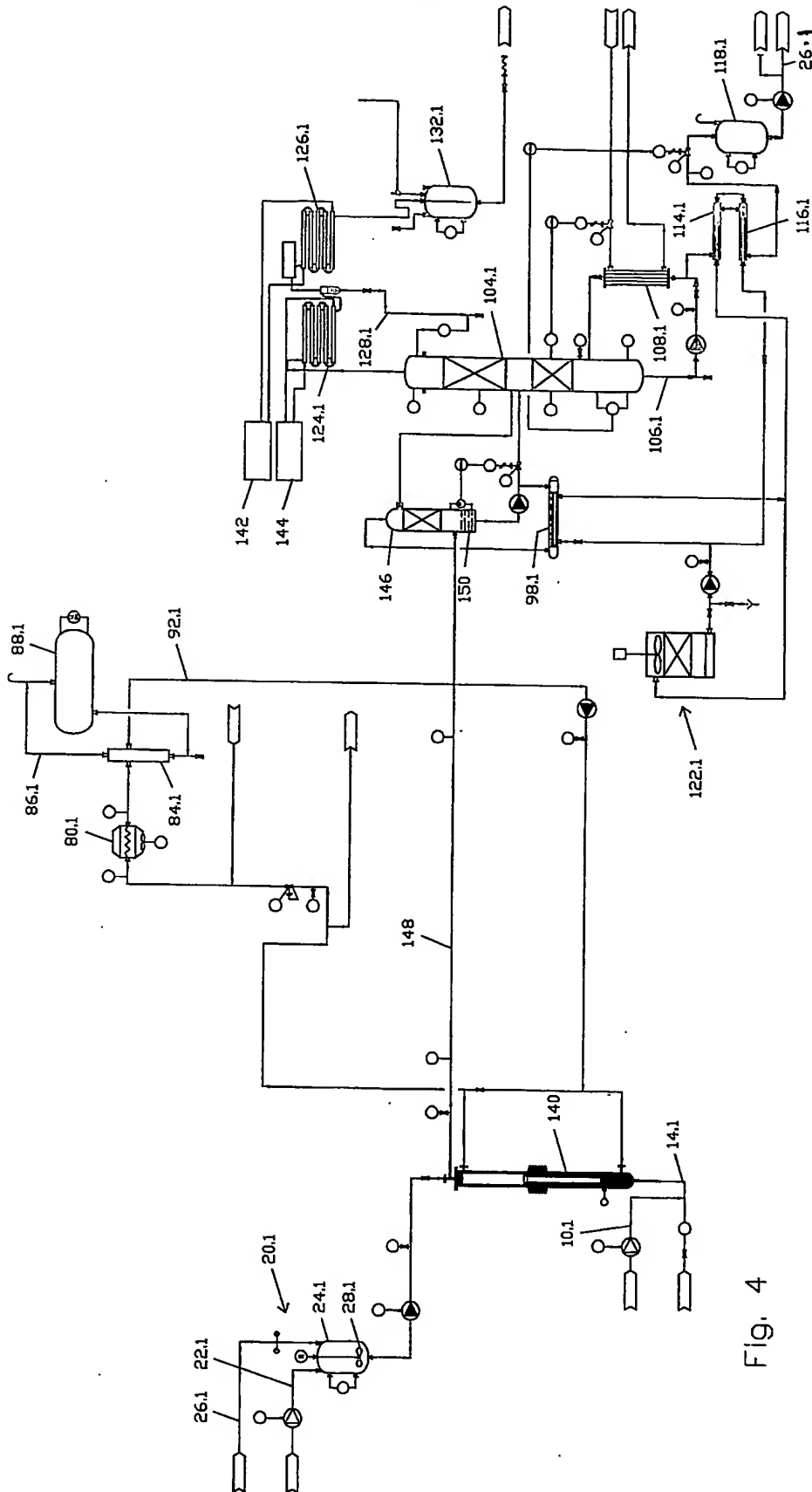


Fig. 4